



RESEARCH ARTICLE

REVISED Corrosion of copper nickel titanium archwire in chlorhexidine, sodium fluoride, and chitosan mouthwashes [version 3; peer review: 3 approved]

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Abstract

Background

Copper (Cu), nickel (Ni), chromium (Cr) ion release, and surface topography change from the orthodontic wire are the initial processes of corrosion that may affect the mechanical properties of the archwire. In this study, we aim to evaluate the effect of CHX, NaF, and chitosan on the corrosion of CuNiTi wire nickel and copper ions released, surface roughness change, and archwire deflection.

Methods

Ninety samples of CuNiTi Tanzo™ archwires were divided into five groups according to their immersion solution: Artificial Saliva, CHX, NaF, CHX-NaF, and chitosan group. Each group was further divided into three subgroups (n=6) corresponding immersion time, *i.e.*, two, four, and six weeks. The corrosion of the samples was analyzed with an atomic absorption spectrophotometer (AAS), scanning electron microscope (SEM), and universal testing machine (UTM).

Results

Open Peer Review

Approval Status

	1	2	3
version 3			
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- Any reports and responses or comments on the article can be found at the end of the article.

The amount of nickel ion releases was increasing, but the copper ion releases were reduced by the time of observations. The highest nickel ion was released in the CHX-NaF group and the lowest in the chitosan group for six-week immersion. It also corresponded to the surface topography by SEM analysis which showed the most extended cracks and deep pits in the CHX-NaF group and a smoother surface in the chitosan group. Copper ion release showed the highest ion release in the NaF group and the lowest release in the chitosan group. The unloading force of CuNiTi archwire deflection remains the same at week two and week four for all mouthwashes.

Conclusion

The use of mouthwashes that contained CHX, NaF, and chitosan could further alter the passive layer and cause higher nickel and copper ion release and increased CuNiTi archwire surface structure porosity. But there is no distinction between mouthwashes to release the unloading force within two until four weeks.

Keywords

CuNiTi archwire, nickel ion release, copper ion release, surface topography, deflection, unloading force, chlorhexidine, and natrium fluoride.

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REVISED Amendments from Version 2

Added:

1. A sentence which recommend to do the intra or inter-reliability test.
2. Mentioned about McDougall's ingredients of artificial saliva, and the mechanical properties of Copper-Nickel-Titanium archwire to the discussion part.

Any further responses from the reviewers can be found at the end of the article**Introduction**

Nickel-titanium alloy archwire offers greater flexibility and resistance to deformation and also exhibits excellent biocompatibility and corrosion resistance (Mitchell, 2013). Further addition of copper (Cu) in Cu nickel (Ni) titanium (Ti) archwires produces a more constant force on the teeth (Singh, 2007; Bhalajhi, 2012; Phulari, 2017). However, in the oral environment, archwires are constantly exposed to various stresses from masticatory forces, loading appliances, temperature fluctuations, and varieties of ingested food and saliva (Chaturvedi and Upadhayay, 2010).

The electrochemical mechanism of corrosion plays an essential role in metal corrosion when in contact with an electrolyte fluid, for example, saliva or mouthwashes. When two different alloys are in contact with a fluid electrolyte, the alloy with lower electrode potential will become the anode and produce oxidation that released several ions into the solution (Anusavice *et al.*, 2012). Corrosion creates two issues; it can alter the physical properties of archwires (Mane *et al.*, 2012; Geramy, Hooshmand and Etezadi, 2017; Doddamani, Ghosh and Tan, 2018) and cause a local or systemic condition due to allergic reactions and biological side effects (Lü *et al.*, 2009; Pazzini *et al.*, 2009; Suárez *et al.*, 2010; Hafez *et al.*, 2011; Castro *et al.*, 2015). The metal products released during corrosion are nickel, chromium, and copper. Nickel is classified as a chemical carcinogen (IARC Working Group, 1990). In addition, it is a powerful medium for an immune reaction which can lead to a hypersensitivity reaction, contact dermatitis, gingivitis, gingival hyperplasia, periodontal stomatitis, periodontitis, burning mouth syndrome, angular cheilitis, cytotoxicity, mutagenic reaction (Hafez *et al.*, 2011; Heravi *et al.*, 2015; Lü *et al.*, 2009). Meanwhile, copper is required by the body, but an excessive amount can produce cytotoxicity in the form of allergies at the point of contact and metal deposits in organs (Farrukh, 2011; Mahalaxmi, 2013).

Deflection of the archwire also plays an essential role in affecting tooth movement during orthodontic treatment (Aghili *et al.*, 2017; Khatri and Mehta, 2014). Deflection of the archwire is defined by the ability of the archwire to transmit forces to the dentoalveolar to promote tooth movement (Parvizi and Rock, 2003; Sarul *et al.*, 2013).

Dental hygienist usually prescribes mouthwashes to patients with low oral hygiene and a high risk of caries to prevent the formation of microbial plaque (Anuwongnukroh *et al.*, 2017; Castro *et al.*, 2015). The use of fluoride mouthwashes helps the enamel layer remineralize and protects it from the acidic environment (Roveri *et al.*, 2009; Sivapriya *et al.*, 2017), but the production of hydrofluoric acid (HF) may have a destructive impact on the archwires. HF degrades the protective oxide layers on the surface, which leads to corrosion (Castro *et al.*, 2015; Hafez *et al.*, 2011; Lü *et al.*, 2009; Marques *et al.*, 2012; Suárez *et al.*, 2010). But there are several published studies on the corrosion resistance of NiTi alloys in saliva and NaF solutions even with increasing concentrations of fluorides (Heravi, Hadi Moayed and Mokhber, 2015; Mirhashemi, Jahangiri and Kharrazifard, 2018; Fatene *et al.*, 2019).

Chlorhexidine also has high effectiveness in preventing the formation of dental plaque and is also effective in decreasing gingival inflammation (Metin-Gürsoy and Uzuner, 2014; Deriaty, Nasution and Yusuf, 2018). Several authors have evaluated a significant lowering in corrosion resistance in stainless steel or NiTi archwires in chlorhexidine mouthwashes compared to other mouthwashes (Danaei *et al.*, 2011; Deriaty *et al.*, 2018; Habar and Tatengkeng, 2020). Several studies also showed degradation in the performance of an elastomeric chain (Sufarnap *et al.*, 2021), and more significant surface corrosion was observed under the scanning electron microscope (SEM) in wires from chlorhexidine mouthwashes (Mane *et al.*, 2012; Doddamani, Ghosh and Tan, 2018; Chitra, Prashantha and Rao, 2020).

Chitosan is a natural polysaccharide resulting from the deacetylation of chitin. Chitosan has a broad antibacterial content and a low level of toxicity, so it is often used as a mouthwash for plaque control (Chen and Chung, n.d.; Fei Liu *et al.*, 2000). A study by Uraz *et al.* showed no significant difference in the use of chitosan mouthwash compared to chlorhexidine mouthwash plus chitosan in decreasing plaque index (Uraz *et al.*, 2012).

Several studies were conducted on the corrosion resistance, deflection, and ion release of NiTi orthodontic archwires in chlorhexidine or fluoride mouthwashes. However, there were limited studies observing nickel ion release, copper ion release, deflection test, and surface structure in mouthwashes containing chlorhexidine and fluoride, and chitosan in CuNiTi archwire, and as such this became the objective of this study. We hypothesized the differences found at each immersion solution at each time observation to the surface structure, deflection, and nickel and copper ion release. This study is a continuation of the [Devi et al. \(2022\)](#) study.

Methods

Samples

The research type was an experimental study using a post-test control design. There were ninety (90) Tanzo (American Orthodontics®) CuNiTi 4cm long archwires, sized 0.016×0.022 inches. Samples were divided into five groups according to the immersion solution, *i.e.*, control group, CHX group, NaF group, CHX-NaF group, and chitosan group. The samples were further divided into three subgroups (n=6) corresponding to the duration of immersion, two, four, and six weeks. The sample size was determined with the formulation from [Irmawantini, 2017](#):

$$(n - 1).(r - 1) \geq 15$$

$$(n - 1).(5 - 1) \geq 15$$

$$4n \geq 19 \quad n = 4.75 \text{ (minimum)}$$

Mouthwash immersion phase

Each group has 18 samples divided into three subgroups (n=6) according to the immersion time: two, four, and six weeks. Group 1: immersed in artificial saliva as a control group (produced by the Oral Dental Hospital of Universitas Sumatera Utara Pharmacies followed by McDougall's recipe of minerals: NaHCO₃, Na₂HPO₄·12H₂O, NaCl, KCl, CaCl₂ anhydrous, MgCl₂ anhydrous, and H₂O) ([Khan et al., 2021](#)); Group 2: immersed in artificial saliva and 0.1% chlorhexidine gluconate mouthwash (Minosep, Minorock, Indonesia; as the CHX group); Group 3: immersed into artificial saliva and 0.05% NaF (Merck KGaA, Darmstadt, Germany, as the NaF group); Group 4: immersed into artificial saliva, 0.05% NaF, and 0.12% chlorhexidine gluconate (PerioKin®; as the CHX-NaF group); Group 5: immersed into artificial saliva and 2% chitosan (prawn shells was formulated at the Laboratory of Research Centre (Faculty of Mathematics and Science, Universitas Sumatera Utara as the chitosan group). Ninety samples were made in total, and they were all incubated at 37°C.

Archwires were simulated in the mouth environment, all samples were immersed in 10 ml saliva within observation time (2,4, and 6 weeks), and mouthwashes were simulated two times a day for one minute. All samples immersed corresponding to the subgroup 2, 4, and 6 weeks; Minosep® mouthwash, 0.05% NaF, PerioKin®, and chitosan mouthwash were added into the test tubes in group 2 to 5 simulated respectively for 28 minutes at two weeks subgroup, 56 minutes at four weeks subgroup, and 84 minutes at six weeks subgroup. The samples were agitated with a vortex for one minute before being incubated. After being immersed at each time point, wires were removed from the solutions, washed with distilled water, and dried for further surface roughness topography analysis with the SEM machine. Test tubes were sealed again with aluminum foil and placed at room temperature to prepare the analysis.

Experiment analysis and measurement phase

The research was conducted at the Faculty of Pharmacies Laboratory, Universitas Sumatera Utara, where the samples were incubated at 37°C; nickel and copper ion release sample's immersed solution were analyzed at Balai Standardisasi dan Pelayanan Jasa Industri (Baristand) Medan using atomic absorption spectrometry (AAS, Shimadzu AA7000); the surface structure of CuNiTi wires was tested with a scanning electron microscope (SEM, Hitachi TM3000, Tabletop Microscope, Japan) at 2000× magnification on three sites at Integrated Research Laboratory- Universitas Sumatera Utara; and deflection test with the Universal Testing Machine (UTM, Tensilon RTF 1350) at the Impact Fracture Research Center (IFRC) Laboratory, Faculty of Engineering, Universitas Sumatera Utara.

The SEM images were taken 3 times for each sample. The Region of interest (ROI) of the images captured within the roughest area. The AAS analysis had been taken one time with the Relative Percent Difference (%RPD) below 5%. While the UTM machine results also came with one-time measurements from the machine. Calibrators had been taken 5 times to get the optimal normal curve. The sample which had improper results or outranged the standard or normal curved had been recalculated. The results needed to analyse with the inter- or intra-reliability tests.

Statistic analysis

Statistical analysis was performed using Statistical Package for Social Science (SPSS) 26.0 edition with Shapiro-Wilk for normality test ($p \leq 0.05$). The data obtained were analyzed statistically using the Kruskal-Wallis test to compare the amount of unloading force, nickel, and copper release in weeks two, four, and six.

Results

Mean levels of nickel and copper released in each group for every time observation were significantly different, and the data are shown in Tables 1 and 2, respectively (Sufarnap, 2022). Scanning electron microscope (SEM) images result after six weeks of immersion (the longest time) of CuNiTi's wire surface topography are shown in Figure 1. The roughness was found in all groups, but the most extended surface defects, such as cracks and pits, were found in the CHX-NaF group, followed by more comprehensive pits in the CHX group compared to another group.

According to the result, nickel ions are increasingly released by the time of observation in all groups. In the beginning, the highest nickel released was at the chitosan group, but it had a slow release. The highest amount of nickel release also corresponded to the surface structure, which was the most prolonged observation in group 4 (CHX-NaF) (Figure 2) (Sufarnap *et al.*, 2022).

Copper ions released showed reduced by the time of observation at all groups. The highest copper released was found in the NaF group for all observation times (Figure 3) (Sufarnap *et al.*, 2022). The last analysis was the mean levels of unloading forces in each group. They are shown in Table 3. Based on the results, there were no significant differences in unloading forces at two weeks and four weeks of all groups but showed a significantly different in six-week group. The data are shown in Table 3 and Figure 4 (Sufarnap *et al.*, 2022).

Table 1. Release of Nickel ion from Copper Nickel Titanium wires at different time intervals.

Solution	Mean (ppm in microgram/liter)±SD		
	Week two	Week four	Week six
Control Group	4.85±0.29	9.85±0.19	12.85±0.13
CHX Group	6.01±0.14	10.16±0.08	13.71±0.09
NaF Group	5.78±0.03	10.03±0.01	13.28±0.01
CHX-NaF Group	7.98±0.38 ^{xx}	11.13±0.12	20.20±0.38
Chitosan Group	8.48±0.37	9.29±0.40	11.15±0.94
p-value	0.001^a	0.001^a	0.001^a

Data from five groups are expressed as mean±standard deviation (SD) (n=10). Superscript letters are significantly different by the *Kruskal Wallis* test ($p < 0.05$). NaF: Sodium Fluoride, CHX: Chlorhexidine, CHX-NaF: Chlorhexidine-Sodium Fluoride.

Table 2. Release of Copper ion from Copper Nickel Titanium wires at different time intervals.

Solution	Mean (ppm in microgram/liter)±SD		
	Week-two	Week-four	Week-six
Control Group	1.62±0.03	1.41±0.04	1.28±0.03
CHX Group	2.38±0.04	2.24±0.08	2.20±0.05
NaF Group	3.89±0.001	3.54±0.001	3.23±0.001
CHX-NaF Group	3.37±0.07	3.06±0.10	3.00±0.06
Chitosan Group	1.07±0.04	1.07±0.04	0.97±0.02
p-value	0.001^a	0.001^a	0.001^a

Data from five groups are expressed as mean±standard deviation (SD) (n=6). Superscript letters are significantly different by the *Kruskal Wallis* test ($p < 0.05$). CHX: Chlorhexidine, CHX-NaF: Chlorhexidine-Sodium Fluoride, Chitosan: Chitosan 2%.

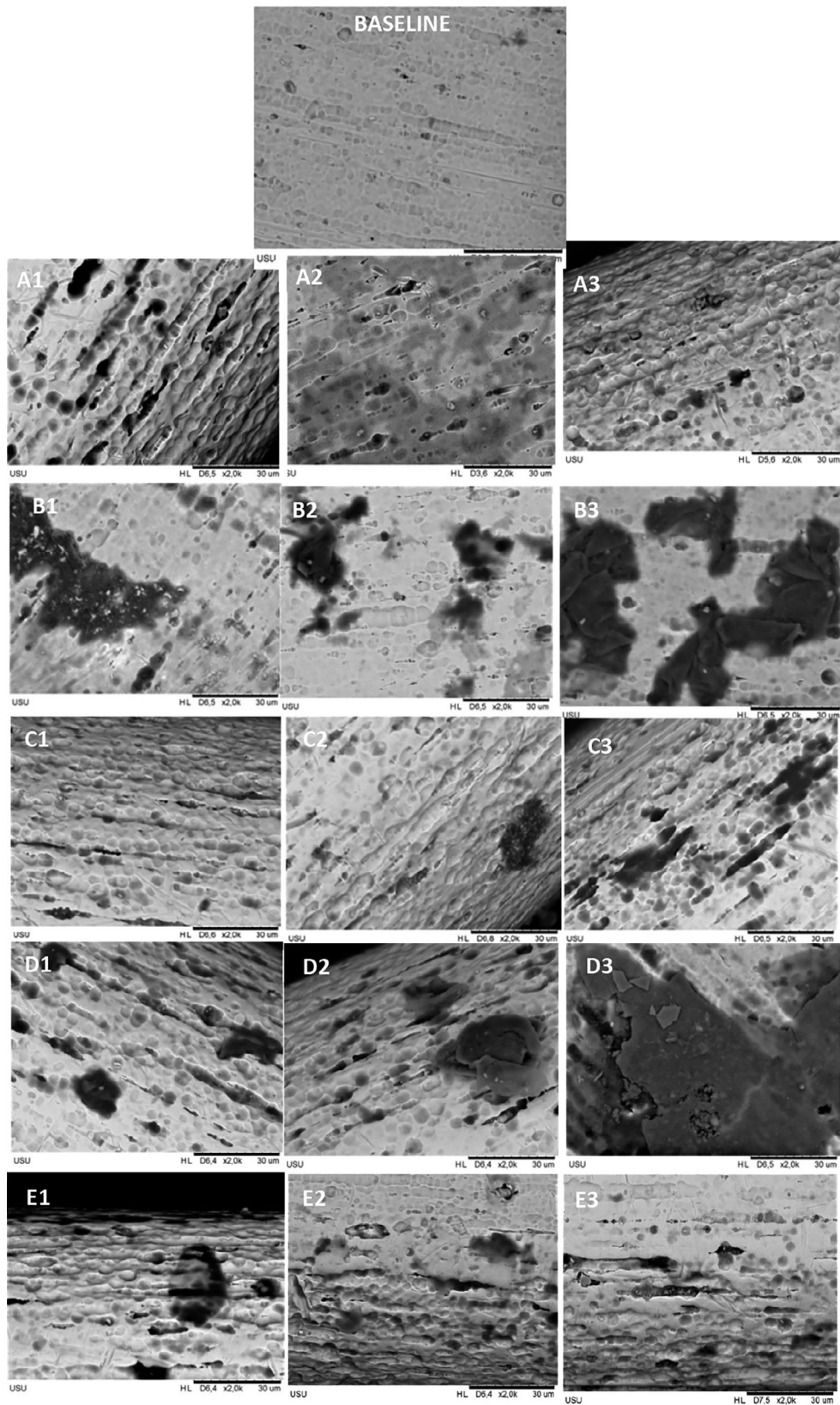


Figure 1. Surface roughness of CuNiTi archwire with SEM in 2000 magnification. Immersed with A. Control Group B. CHX Group C. NaF Group D. CHX+NaF Group E. Chitosan Group. (1,2,3 represent 2, 4, and 6 weeks).

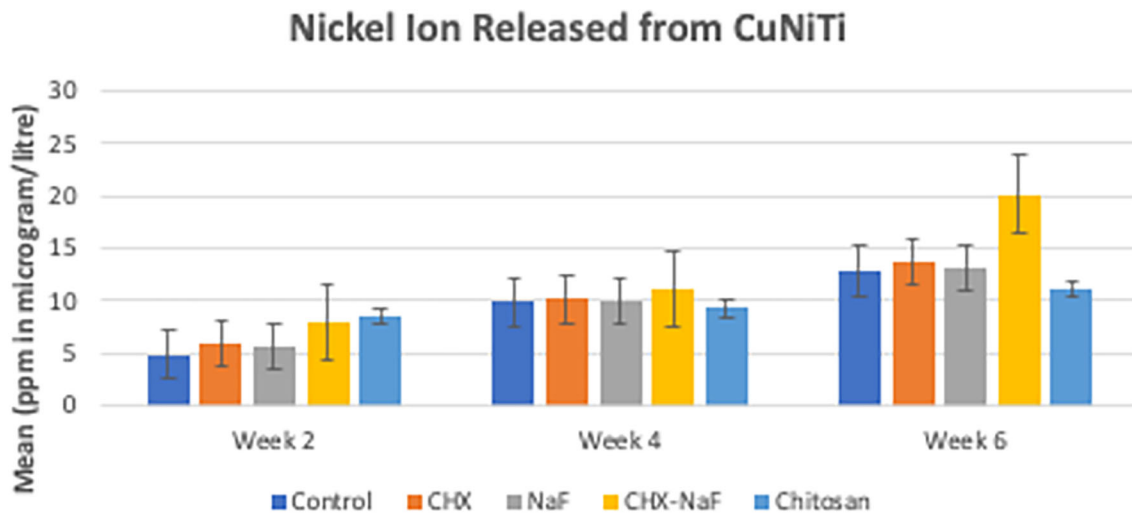


Figure 2. Comparison of Nickel released (ppm in $\mu\text{g/L}$) from CuNiTi wires at different time intervals. Abbreviations: NaF: Sodium Fluoride; CHX: Chlorhexidine; CHX-NaF: Chlorhexidine-Sodium Fluoride.

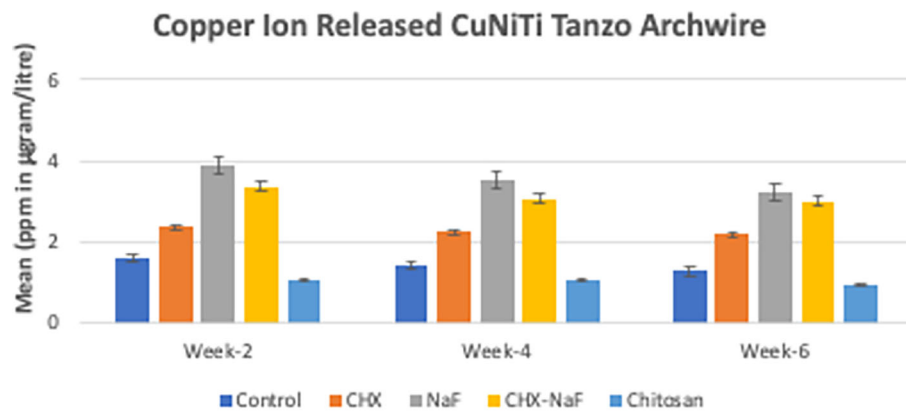


Figure 3. Comparison of Copper released (ppm in $\mu\text{g/L}$) from CuNiTi wires at different time intervals. Abbreviations: NaF: Sodium Fluoride; CHX: Chlorhexidine; CHX-NaF: Chlorhexidine-Sodium Fluoride.

Table 3. Unloading forces from Copper Nickel Titanium wires at different time intervals (the baseline value was 45.049 Newton (N)).

Solution	Mean (N) \pm SD			p-value**
	Week two	Week four	Week six	
Control Group	47.639 \pm 2.1234	45.936 \pm 1.7143	47.207 \pm 2.2875	0.262
CHX Group	47.798 \pm 3.7015	42.532 \pm 8.8856	45.864 \pm 2.0239	0.532
NaF Group	47.95 \pm 2.32	48.89 \pm 1.23	48.14 \pm 1.34	0.623
CHX-NaF Group	50.141 \pm 1.3432	46.546 \pm 2.7308	46.224 \pm 1.1263	0.010
Chitosan Group	49.349 \pm 0.7325	45.428 \pm 1.7099	48.621 \pm 0.8625	0.003
p-value*	0.195	0.086	0.023	

Data from five groups are expressed as mean \pm standard deviation (SD) (n=6).
 *p-value between mouthwashes; **p-value between Kruskal Wallis test (p<0.05).
 NaF: Sodium Fluoride, CHX: Chlorhexidine, CHX-NaF: Chlorhexidine-Sodium Fluoride.

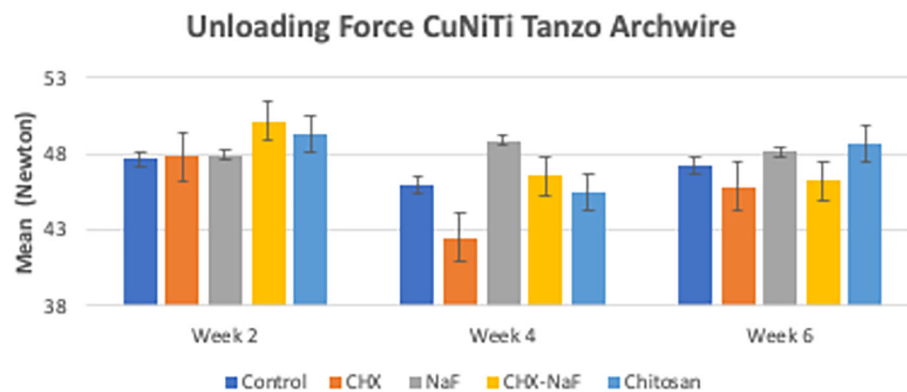


Figure 4. Comparison of unloading deflection force from CuNiTi wires at different time intervals. Abbreviations: NaF: Sodium Fluoride; CHX: Chlorhexidine; CHX-NaF: Chlorhexidine-Sodium Fluoride.

Discussion

The highest amount of nickel ion was 0.2020 mg from the CHX-NaF group at the six-week group, and the highest amount of copper ion was 0.03377 mg from the CHX-NaF group at week two. This showed the highest concentration of both ions was still below the warned concentration limit. The average amount of nickel intake obtained from food is 300 µg–500 µg/day (Alarifi *et al.*, 2013; Milheiro *et al.*, 2016). While a nickel concentration of 600 µg–2500 µg can induce an allergic reaction, and a copper concentration of 10 ppm can cause a cytotoxic reaction (Danaei *et al.*, 2011; Milheiro *et al.*, 2016). The nickel and copper ions in this study were still released in the artificial saliva (control group), although it had the least amount compared to the other group. The mechanical properties of CuNiTi archwire consists 46,87% of nickel (NiO), 43,70% of Titanium (TiO₂) and 6,72% of copper (CuO) (Gravina *et al.*, 2014). These properties explained why the nickel ion released more than the copper ion release within this CuNiTi archwire.

Chlorhexidine used for a long time can generate Reactive Oxygen Species (ROS), the primary agent responsible for endogenous DNA damage (Septiani and Auerkari, 2020). Nik *et al.* and Omidkhoda *et al.* found that CHX immersion had no significant effect on NiTi archwire surface roughness, corrosion, and frictional resistance (Danaei *et al.*, 2011; Nik *et al.*, 2013). But some authors found a significant decrease in corrosion resistance due to CHX mouthwashes (Danaei *et al.*, 2011; Deriaty *et al.*, 2018; Habar and Tatengkeng, 2020), and some authors found more significant surface corrosion with SEM analysis (Mane *et al.*, 2012; Doddamani, Ghosh and Tan, 2018; Chitra, Prashantha and Rao, 2020). This study also found the surface topography showed that the CHX group had rougher and more pitted surfaces compared to the control group or baseline topography.

In six weeks of immersion, CuNiTi wire in the CHX-NaF group has the highest amount of nickel ions released and in the NaF group, more copper ions were released than the other group. The unloading force difference is also significantly seen in the CHX-NaF and chitosan groups within different immersion durations. As a result, there were changes in CuNiTi mechanical properties after immersion of CHX-NaF, NaF, and chitosan for six weeks.

NaF content in perioKin® (CHX-NaF) mouthwash can increase the metal ions released from the CuNiTi orthodontic wire. Heravi *et al.* found that the NaF content in mouthwash solution would decrease the corrosion resistance of NiTi and CuNiTi wires as the NaF concentration increased (Heravi, Hadi Moayed and Mokhber, 2015; Fatene *et al.*, 2019). Sabah and Jarjees also reported that the interaction between fluoride and titanium might cause destruction to the metal coating of the archwires and degrade the mechanical properties (Sabah, Jarjees and Awni, 2011).

The immersion of chitosan showed significant differences in Nickel and copper ion release, surface topography, and unloading force. Chitosan mouthwash has been used due to its antibacterial effect and lack of side effects. Uraz *et al.* has reported that chitosan 2% mouthwash had no significant difference compared to CHX 0,2% in reducing plaque index and gingivitis (Uraz *et al.*, 2012).

The highest nickel ion released was found in the chitosan group at week two, but it increased slowly and steadily compared to other groups, and it lasted with the lowest nickel released for the longest immersion time (six-week immersion). The same result with copper ions released. It was found that the chitosan had the lowest amount of ions released from week two until six weeks. Unfortunately, nothing was found about the chitosan particles used as an

inhibitory of corrosion for orthodontic archwire, but Putri *et al.* researched orthodontic mini-implant immersed in chitosan mouthwashes for the surface topography found that the mini implant immersed with 1.5% of chitosan mouthwash had a smoother surface compared to chlorhexidine and Sodium Fluoride mouthwash. The CuNiTi archwire surface topography in this study found that the roughness of the chitosan group also had a smoother area compared to CHX and CHX-NaF groups (Putri *et al.*, 2021).

Furlan *et al.* mentioned that there were differences in the amount of nickel and copper ions released in several types of CuNiTi wire immersed in a neutral solution and an acidic solution, which were greater in the acidic solution (Furlan *et al.*, 2018). The main limitation of this study was that it did not analyze the pH changes within each immersion solution and each observation time. This study was also done under the static condition *in vitro*. Further research is needed to determine the situation with *in vivo* experiments. The surface structure should also be better analyzed with an advanced equipment, *i.e.*, atomic force microscopy (AFC) so that we could measure the quantity of the surface by its roughness. This will allow us to better understand the physical properties of CuNiTi wire. The inter- or intra-reliability test should appropriately had been suggested for several samples.

Conclusions

CuNiTi wire had an increase of nickel and reduced copper ion release parallel to the increase of immersion time, but the deflection of the wire did not show any significant differences between mouthwashes. However, after six weeks of immersion, the amount of Nickel and copper ions released was still within the safe limit. The surface roughness of CuNiTi archwire topography showed that the CHX-NaF group had the most extended cracks and deep pits. The unloading force of the CuNiTi archwire deflection remains the same at week two and week four for all mouthwashes.

Data availability

Underlying data

OSF: Data of Corrosion of CopperNickelTitanium Archwire in Chlorhexidine, Sodium Fluoride and Chitosan Mouthwashes, <https://doi.org/10.17605/OSF.IO/5QB8E> (Sufarnap, 2022).

This project contains the following underlying data:

- Copper Ion Release of CuNiTi Tanzo Archwires.pdf
- Deflection of CuNiTi Tanzo Archwires.pdf
- Nickel Ion Ni Release of CuNiTi Tanzo Archwire.pdf
- SEM Surface topography of Tanzo AW (folder containing all raw SEM images)

Extended data

OSF: Data of Corrosion of CopperNickelTitanium Archwire in Chlorhexidine, Sodium Fluoride and Chitosan Mouthwashes, <https://doi.org/10.17605/OSF.IO/5QB8E> (Sufarnap, 2022).

This project contains the following extended data:

- Figure 1, 2,4. Graph of Ni-Cu Ion Release and Deflection of Tanzo CuNiTi Archwires.pdf
- Figure 3. Surface Analysis of CuNiTi Tanzo Archwire (SEM analysis).jpeg

Data are available under the terms of the [Creative Commons Zero “No rights reserved” data waiver](https://creativecommons.org/licenses/by/4.0/) (CC0 1.0 Public domain dedication).

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Andrzej Zielinski

Gdansk University of Technology, Gdańsk, Poland

After considering all the changes and limitations in the performed research I have no further comments. It is a perfect piece of research, well-written, and it will certainly be of interest. I admire the hard work of the authors in improving the manuscript.

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Biomaterials engineering

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard.

Reviewer Report 04 June 2024

<https://doi.org/10.5256/f1000research.163146.r284847>

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Asma Ashari 

Department of Family Oral Health, Faculty of Dentistry, Universiti Kebangsaan Malaysia, Kuala Lumpur, Malaysia

Thank you and no further comment.

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Orthodontics, clear aligners

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard.

Version 2

Reviewer Report 04 June 2024

<https://doi.org/10.5256/f1000research.145994.r167825>

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Asma Ashari 

Department of Family Oral Health, Faculty of Dentistry, Universiti Kebangsaan Malaysia, Kuala Lumpur, Malaysia

Under the heading "Mouthwash immersion phase" Please edit the second paragraph:

"Archwires were simulated in the mouth environment, all samples were immersed in 10 ml saliva within observation time (2,4, and 6 weeks), and mouthwashes were simulated two times a day for one minute. **One minute is the recommended time as per manufacturer's instructions.**"

Under the heading "Experimental analysis and measurement phase" can rephrase second paragraph:

"One researcher (Initials) with more than 3 years of experience in AAS, UTM and SEM machines did the measurement of all the samples. The region of Interest (ROI)....."

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Orthodontics, clear aligners

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Reviewer Report 21 February 2024

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Andrzej Zielinski

Gdansk University of Technology, Gdańsk, Poland

I have three remarks:

1. All tests have been performed in saliva or solution containing saliva. It is obligatory to repeat the tests to know the chemical composition of this artificial, presumably, saliva.
2. The results are positive for all users of archeries, but from a scientific point of view, I am not fully satisfied. Please attempt, it might be even speculative and short, why nickel ions are released relatively quickly, and copper ions are not, scarcely dependent on chemical solution composition.
- 3, Twice words are unnecessarily started with capital letters: Chitosan (page 3, bottom of the page), and Nickel (page 9, Conclusions)

Is the work clearly and accurately presented and does it cite the current literature?

Yes

Is the study design appropriate and is the work technically sound?

Yes

Are sufficient details of methods and analysis provided to allow replication by others?

No

If applicable, is the statistical analysis and its interpretation appropriate?

Yes

Are all the source data underlying the results available to ensure full reproducibility?

Yes

Are the conclusions drawn adequately supported by the results?

Yes

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Biomaterials engineering

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Author Response 27 May 2024

Erliera Sufarnap

Dear Professor Andrzej Zielinski,

Thank you for allowing us to revised the manuscript. We appreciate the time and effort that you gave and provided us with many valuable comments and feedback to make this manuscript become more clear and great. We were trying our best efforts to make revisions regarding your inquiries.

The following are our comprehensive three comment's answers after revised:

1. All tests have been performed in saliva or solution containing saliva. It is obligatory to repeat the tests to know the chemical composition of this artificial, presumably, saliva.

Answers:

Thank you Professor Zielinski for the inquiry.

The study had been performed with artificial solution presumably most closely to the saliva's compositions. The solution derived with McDougall's recipe of minerals with exact ingredient and concentrations which consists of NaHCO_3 , $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$, NaCl , KCl , CaCl_2 anhydrous, MgCl_2 anhydrous, and H_2O . We've found the original McDougall's journal about this recipe in detail, but from the year 1947 presumably it had been cited in many journals, and the newest journal found by Khan et al. 2021 which we were attached to the manuscript. Most journal had this recipes made by pharmacist with their own laboratory until now without any modification and also in our hospital pharmacy.

We were terribly sorry we didn't mention this in the manuscript. We've already added it to the manuscript at the "Methods" parts, at Group 1. Here, we also attached the table, cited originally from McDougall's journal 1947.

[Reference Link to Attached table](#)

2. The results are positive for all users of archeries, but from a scientific point of view, I am not fully satisfied. Please attempt, it might be even speculative and short, why nickel ions are released relatively quickly and copper ions are not, scarcely dependent on chemical solution composition

Answers:

The addition of copper (Co) causes a more constant force distribution during activation since the copper has it's heat conductor function within certain temperature which considered in the mouth. It also gives greater flexibility and exhibits corrosion resistance (Mitchell 2013, Gravina et al, 2014).

The mechanical properties of CuNiTi archwire consists 46,87% of nickel (NiO), 43,70% of Titanium (TiO_2) and 6,72% of copper (CuO) (Gravina et al. 2014). These properties could explained why the nickel ion released were greater than the copper ion release due to Ni properties were greater than Co.

The chemical solution composition and the acidity would also be a predisposition to greater ion released, for example that the solution which contained a fluoride and also the acid condition of the solution would gave more tendency to higher ion released compared to

controls which I already described at the last paragraph from discussion part.
Thank you Professor for the inquiries, I explained about this in discussion session to make the results and discussion more clear.

3. Twice words are unnecessarily started with capital letters: Chitosan (page 3, bottom of the page), and Nickel (page 9, Conclusions)

Thank you very much, dear Professor. I corrected the typos in my manuscript by de-capitalizing the letter "chitosan" in many words.

Competing Interests: There are no competing of interest

Reviewer Report 11 January 2024

<https://doi.org/10.5256/f1000research.145994.r230212>

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SHAZA HAMMAD

Mansoura University, Mansoura, Dakahlia Governorate, Egypt

The manuscript was conducted to answer an important issue concerning the effect of CHX, NaF, and chitosan on the corrosion of CuNiTi wire nickel and copper ions released, surface roughness change, and archwire deflection..

The authors modified the manuscript according to previous reviewer's comments.

No need for this sentence" All the laboratory assistants at each laboratory have a Master's degree with above three to seven years of work experience", instead, all is needed is to mention the inter and intra- reliability tests.

Is the work clearly and accurately presented and does it cite the current literature?

Yes

Is the study design appropriate and is the work technically sound?

Yes

Are sufficient details of methods and analysis provided to allow replication by others?

Yes

If applicable, is the statistical analysis and its interpretation appropriate?

Yes

Are all the source data underlying the results available to ensure full reproducibility?

No source data required

Are the conclusions drawn adequately supported by the results?

Yes

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Orthodontic techniques, materials and appliances.

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard.

Author Response 26 Feb 2024

Erliera Sufarnap

Dear Professor Hammad

Thank you for approving our manuscript in F1000Research. We sincerely appreciate you, as the reviewer, who provided your time and effort in giving some feedback to the manuscript. We are very honored and grateful for the insightful comments and valuable improvements to our paper that you gave us. We have already revised and incorporated your inquiries, which we revised in the chapter "Experiment analysis and measurement phase" chapter in the last sentence and the discussion section. These are our revisions. Thank you very much, Prof Hammad.

Our best regards from Indonesia.

Competing Interests: No competing interests were disclosed.

Version 1

Reviewer Report 10 March 2023

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**Asma Ashari**

Department of Family Oral Health, Faculty of Dentistry, Universiti Kebangsaan Malaysia, Kuala Lumpur, Malaysia

An interesting study, although it would benefit from a sample size calculation (if no sample size, can mention it as a pilot study), it would add to the strength of the results, since the sample is

already quite large.

Who did the measurements and who scanned the samples? Are they qualified? Was it a single researcher or multiple? Are they calibrated? Any Intra Class Correlation Coefficient to assess intra and inter-reliability.

"All samples were immersed in 10ml saline within observation time" what does observation time mean? Please elaborate.

The mouthwashes were immersed for one minute twice a day, although in practice is it not usually advice to do it for 30 seconds? And usually patients do it for less than that. Therefore the effects of this study are maybe twice as much as what you would expect clinically.

Mouthwashes are swished around the mouth, not immersed, again this may affect the clinical application.

Shouldn't the wires be washed with saliva instead of with distilled water? since mouthwashes should not be rinsed after gargling.

Is the work clearly and accurately presented and does it cite the current literature?

Yes

Is the study design appropriate and is the work technically sound?

Partly

Are sufficient details of methods and analysis provided to allow replication by others?

Partly

If applicable, is the statistical analysis and its interpretation appropriate?

Partly

Are all the source data underlying the results available to ensure full reproducibility?

Partly

Are the conclusions drawn adequately supported by the results?

Partly

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Orthodontics, clear aligners

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Author Response 22 Mar 2023

Erliera Sufarnap

Thank you very much for your kind attention to the wonderful insightful comments on our manuscript. This manuscript became very complete based on your pieces of advice in many chapters. We have revised the manuscript according to some inquiries you have suggested. Several additional sentences needed to be added based on the reviewer's suggestions, which we already provided to consolidate the new sample size formulation.

The following is our comprehensive comments on the review after revision:

1. An interesting study, although it would benefit from a sample size calculation (if no sample size, can mention it as a pilot study), it would add to the strength of the results, since the sample is already quite large. The sample size was conducted by using the Federer Technique in our references by Irmawartini as she stated in her book. We added the formulation to the manuscript.

2. Who did the measurements and who scanned the samples? Are they qualified? Was it a single researcher or multiple? Are they calibrated? Any Intra Class Correlation Coefficient to assess intra and inter-reliability.

a. The measurement and the scanning process were done by one qualified laboratory assistant (Master's degree) in each lab. Their work experience is above 3 years for AAS and UTM machines and 7 years for SEM machines.

b. Calibration

- The SEM machine calibrates twice in a year interval.

- The AAS machine calibrates once a year interval.

- The UTM machines were calibrated before each study starts for each day of measurement. The laboratory has 1 to 3 times studies each year.

c. - The SEM images were taken 3 times for each sample. The SEM images couldn't be measured objectively. The Region of interest (ROI) of the images captured within the roughest area.

- The AAS analysis had been taken one time with the Relative Percent Difference (%RPD) below 5%

- The UTM machine results also came from one-time measurements from the machine. Calibration had been taken 5 times to get the optimal normal curve. The sample which had improper results were recalculated. Due to the 3 time images captured from SEM analysis, the %RPD below 5% for Ions released measurements and the 5 times calibrator had been taken to get the normal curve from the UTM machine measurement, all laboratory assistants were convinced that there was no need any intra or the inter reliability analysis for those inquiries.

Thank you, Doctor, but we added the above explanation in the manuscript for each part of the analysis in the M&M section.

3. "All samples were immersed in 10ml saliva within observation time" what does observation time mean? Please elaborate.

Observation time means the time as each subgroup time. All samples immersed corresponds to subgroup 2, 4, and 6 weeks. Samples were immersed in 10ml saliva within each observation time, acting as a simulation of the daily patient had the archwire within the mouth environment.

This was our novel method to have a proper simulation of the mouth condition. The archwire did not immerse with only the mouthwash itself which commonly had the high detectable results.

This method also prevented bad results coming with undetectable measurements if only a short immersed time of each mouthwash at each observation time, 2, 4, and 6 weeks coincidence to 28, 56, and 84 minutes of immersion with the simulation of mouthwash use in 1 minute twice a day.

4. The mouthwashes were immersed for one minute twice a day, although in practice is it not usually advised to do it for 30 seconds? And usually, patients do it for less than that. Therefore the effects of this study are maybe twice as much as what you would expect clinically.

Based on our mouthwash prescription, it is instructed to do the mouthwash twice a day for 1 minute. Therefore, we conduct the immersion time based on it. The condition in which the patient does it for less than the prescribed immersion time is uncontrollable. As a result, we decided on the immersion time as the mouthwash company prescribed.

5. Mouthwashes are swished around the mouth, not immersed, again this may affect the clinical application.

Yes, indeed. Mouthwashes are swished around and mixed with saliva in the patient's mouth. However, we are using the same amount of mouthwash as it is usually used which is 10mL and mixed with saliva. In addition, the use of an agitator before immersion in the mouthwashes makes the solution homogenous. The samples were once again agitated before the archwire was removed for further analysis. By doing these, hopefully, won't affect the clinical application.

6. Shouldn't the wires be washed with saliva instead of with distilled water? since mouthwashes should not be rinsed after gargling.

While the observation time was ended, the wires have been removed and washed with distilled water to continue for another analysis (surface roughness topography with SEM machine), so that all biological or any material which could contaminate the surface image should be eliminated.

Competing Interests: There were no competing interests were disclosed.

Reviewer Response 09 Mar 2024

Asma Ashari

I now approve this report

Competing Interests: No competing interests were disclosed.

Comments on this article

Version 1

Author Response 15 Apr 2023

Erliera Sufarnap

Dear Readers,

Please cite our manuscript for all readers who read our explanation and will use our methods, especially in the simulation of the mouth condition. Thank you for being so understanding—regards by all authors.

Competing Interests: No competing interests were disclosed.

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